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Issued December 26, 1912.

United States Department of Agriculture,

BUREAU OF CHEMISTRY—Circular No. 108.

R. E. DOOLITTLE, Acting Chief of Bureau.

EXTRACTS FROM THE PROCEEDINGS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1912.[1]

REPORTS ON THE RECOMMENDATIONS OF REFEREES.

COMMITTEE A.—J. P. STREET, CHAIRMAN.

(Nitrogen, potash, phosphoric acid. soils, inorganic plant constituents, insecticides, water, and separation of nitrogenous bodies (vegetable proteins).)

NITROGEN.

It is recommended—

(1) That the association continue the study of the neutral and alkaline permanganate methods for organic nitrogen availability.

Approved for further study.

(2) That the suggested method for determining nitrogen in nitrates (Bul. 152, p. 28; Cir. 90, p. 2) be further studied.

Approved for further study.

POTASH.

It is recommended—

(1) That the proposed modification of the official method (Bul. 152, p. 41) be adopted as official.

Adopted, final action.

(2) That the referee for next year study by pot or plot experiments the relative availability of the potash from different sources, comparing especially the potash of known organic origin with inorganic forms.

Approved.

PHOSPHORIC ACID.

It is recommended—

(1) That further work be done next year on the methods for basic slags with the methods and samples used by the referee this year.

Approved.

SOILS.

It is recommended—

(1) That the Rather modification (Bul. 152, p. 52; Cir. 90, p. 3), precipitating the clay in the humus by means of ammonium carbonate, be studied further.

Approved for further study.

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[1] An advance circular giving the recommendations of referees as adopted and other action affecting the work of 1913, together with the officers, referees, and committees appointed.

(2) That the study of methods of extracting humus and methods for acidity of soils be continued.

Approved.

INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the referee for next year be instructed to study further the Schreiber method (Cir. 56) for total sulphur.

Approved.

(2) That further cooperative study of the oxalate method for iron and aluminum be discontinued.

Approved.

(3) That the molybdate method extended to the determination of calcium as oxalate and magnesium as ammonium magnesium phosphate (Bul. 152, p. 62; Cir. 90, p. 5) be further studied.

Approved for further study.

(4) That the molybdate method for the separation of iron and aluminum in an ash solution (Bul. 152, p. 61; Cir. 90, p. 4) be adopted as official.

Adopted, final action.

INSECTICIDES.

It is recommended—

(1) That the chromate method (Method II) for total lead oxid in lead arsenate (Bul. 152, p. 68) be adopted as official.

Adopted, final action.

(2) That further action on recommendations (3), (4), (5), and (6) of the referee for 1911 be deferred pending further study.

Approved. (Available for final action, 1913.)

These recommendations are as follows:

(3) That the method for total sulphur in lime-sulphur solutions (Bul. 107, Rev., p. 34) be changed as follows: Under "2. Determination," line 1, after "Measure," insert "and accurately weigh"; after "sample" strike out "in" and insert a comma and the words "transfer to"; and that the method as thus changed be recommended for final adoption as official in 1912.

(4) That the gravimetric method for sulphur as sulphids and polysulphids in lime-sulphur solutions, as given in the referee's report, be adopted as official.

(5) That the volumetric method for sulphur occurring as thiosulphate in lime-sulphur solutions, as given in the referee's report, be adopted as official.

(6) That the method given (by the referee) for sulphur occurring as sulphates and sulphites in lime-sulphur solutions be changed as follows: Strike out "heat to boiling," etc., to end of paragraph, and substitute "warm on steam bath, precipitate with barium chlorid solution, stirring vigorously for several minutes, let stand in the cold overnight, filter, and obtain weight of barium sulphate. From this weight calculate sulphur," and that the method as changed be adopted as official.

(3) That the provisional methods for the analysis of lead arsenate (Bul. 107, Rev., p. 239) be changed in accordance with recommendation 7 of the referee in 1910 (methods (a) and (b) substituted and (c) and (d) added, as given in Bul. 137, p. 38), and, as changed, be adopted as official.

Adopted, final action.

(4) That the following methods, (a), (b), (c), (d), (e), for the analysis of lime-sulphur solutions be studied further next year in comparison with the methods as reported by the referee for 1911.

Approved for further study.

(a) *Total sulphur solution.*—Weigh accurately 5 to 10 grams of the solution, depending upon the concentration. Make to 100 cc with carbon-dioxid-free water and use 10 cc aliquots for each determination. (In case of concentrates

5 grams should be made to 200 cc and 10 cc aliquots should be used.) Transfer the aliquots to a wide-neck Erlenmeyer flask, add 3 grams of sodium peroxid, cover immediately with a watch glass, and heat on a steam bath 5 or 6 minutes after shaking it thoroughly. Dilute somewhat and add 10 cc of concentrated hydrochloric acid. Make to about 140 cc, boil a few minutes to drive off the dissolved gases, then add, drop by drop, at the rate of 5 cc per minute, 10 cc of 10 per cent solution of barium chlorid. The solution must not be shaken during the addition of the barium chlorid. Set aside to cool. The precipitation may be made with equal accuracy in the cold, provided 5 per cent barium chlorid solution is used instead of 10 per cent. If precipitated in the cold, it should stand overnight before filtering. (See Folin, J. Biol. Chem., 1905, *1*: 131.) The precipitated barium sulphate should be heated in a closed crucible at very low heat until the filter is thoroughly charred. Partially remove the lid and continue the ignition gradually until constant weight with the full heat of the Bunsen flame. Calculate sulphur from weight of barium sulphate.

(b) *Thiosulphate sulphur.*—Dilute 10 cc of solution prepared for total sulphur to about 30 cc in a wide-neck Erlenmeyer flask and titrate with tenth-normal iodin until the yellow color of the polysulphids just disappears. This point is easily determined, provided the flask is surrounded by white surfaces over a white plate. Titrate very carefully near the disappearance of the color, with thorough shaking. Note the volume of iodin used, continue the above titration, letting the iodin act as its own indicator until a small drop produces a slight permanent coloration. The additional iodin represents thiosulphate sulphur according to the following reaction: $2\ CaS_2O_3+I_2=CaI_2+CaS_4O_6$ (1 cc N/10 I=0.0064 S).

The iodin used to discharge the color of the polysulphids determines the mono-sulphur equivalent as follows: $I_2=S$ (1 cc N/10 I=0.0016 S).

(c) *Sulphate and sulphite sulphur.*—To the filtrate with one washing from the thiosulphate determination add several drops of hydrochloric acid and precipitate in the cold with about 5 cc of 5 per cent barium chlorid. Let stand overnight in the cold. From the weight of barium sulphate calculate the sulphate and sulphite (now sulphate) sulphur.

(d) *Sulphid sulphur* (direct determination).—Place the filtered and washed sulphur from the thiosulphate determination with the filter paper in a wide-neck Erlenmeyer and dissolve the sulphur in 15 cc (1 to 3) sodium hydroxid (do not use potassium hydroxid), heating on steam or water bath until the sulphur dissolves (do not boil). It requires from 1 to 1½ hours on steam bath to dissolve the sulphur. Keep the flask covered and shake gently a few times during the digestion to get the sulphur off the sides. Oxidize and complete the determination as in total sulphur.

(Note: A blank must be run on the sodium hydroxid used and correction made therefor.)

The difference between the sum of the thiosulphate and the sulphate sulphur and the total sulphur is sulphid sulphur.

(e) *Total lime (CaO) in solution.*—Oxidize 10 cc of solution prepared for total sulphur as for total sulphur, make acid in the same way, dilute to about 50 cc and boil a few minutes to drive off dissolved gases, make faintly alkaline with ammonium hydroxid, and if a precipitate occurs filter and wash. Precipitate with ammonium oxalate in the usual way and weigh the total lime.

(5) That the referee for next year study the following method for determining water-soluble arsenic in lead arsenate in comparison with the present provisional method extracting for 10 days.

Approved.

Water-soluble arsenic—Weigh to 0.01 gram about 2 grams of paste. Place in a tightly stoppered bottle with 200 cc of carbon-dioxid-free water per gram, put in a mechanical shaker and shake for 3 hours and let stand 2 hours and filter; or, instead of using the mechanical shaker, shake every half hour or as often as possible during a working day. Let stand overnight and filter. Use 200 cc of the clear filtrate for the determination. Add 0.5 cc of sulphuric acid and proceed as directed under water-soluble arsenic oxid, Bulletin 107, Revised, page 240. It is important that the solution shall be perfectly clear and the titrations carefully made. Make corrections for amount of iodin to produce the same color, using same chemicals and volumes.

WATER.

It is recommended—

(1) That the phenol sulphonic acid method for nitrogen in the form of nitrates be adopted as an official method.

Approved for final action as official in 1913.

Phenol sulphonic acid method for nitrates.—Add standard silver sulphate free from nitrate (4.3969 grams per liter, 1 cc equals 1 mg of chlorin) precipitating all but about 0.5 mg of the chlorin. Heat to boiling, allow to settle, or add a little alumina cream, filter and wash with small amounts of hot water. Evaporate the filtrate to dryness in porcelain; when cold add 2 cc of phenol disulphonic acid reagent (prepared by dissolving 25 grams of pure white phenol in 150 cc of c. p. sulphuric acid concentrated and 75 cc of fuming sulphuric acid 13 per cent SO_3 and heating at 100° C. for 2 hours), rubbing with a glass rod to insure intimate contact. Dilute with distilled water and add slowly ammonium hydroxid until the maximum color is developed. Transfer to a colorimetric cylinder, filtering if necessary, and compare with a standard potassium nitrate solution (containing 0.01 mg of nitrogen as nitrate in 1 cc) which has been treated in like manner with phenol disulphonic acid reagent. Compare in the usual manner and record as nitrogen in the form of nitrates.

(Note: A portion of the standard potassium nitrate solution may be evaporated, the residue treated with phenol disulphonic acid, taken up with water, and made up to a definite volume. This solution will keep, and standards for comparison may be prepared by adding ammonia to measured volumes of it.)

(2) That the reduction method for nitrogen in the form of nitrates be adopted as an optional method.

Approved for final action in 1913.

Reduction method for nitrates.—(Reagents: (1) Sodium or potassium hydroxid solution. Dissolve 250 grams of the purest hydroxid obtainable in 1.25 liters of distilled water and boil down to 1 liter. (2) Aluminum foil. Use strips about 5 cm long, 0.012 mm thick, and of such a width that each strip weighs about 0.35 gram.)

Put 10 cc of the sample in a Nessler tube and make up to exactly 50 cc; add 5 cc of sodium hydroxid solution and a strip of aluminum foil. Close the mouth of the Nessler tube with a rubber stopper carrying a U-shaped glass tube of such length that the outlet can be placed in a second test tube containing a small amount of diluted hydrochloric acid, which serves as a trap to catch any ammonia which may escape. Allow to stand at room temperature for 12 hours or more until reduction is complete. Transfer contents of tube to a Kjeldahl flask and distill. Cool the distillates and Nesslerize in the usual way. Also Nesslerize the solution in traps.

(3) That the method for strontium be further studied.

Approved for further study.

Strontium.—Evaporate such a quantity of the water that the weight of the calcium oxid will be from 0.1 to 0.6 gram, remove silica, iron, and aluminum in the usual way and make to from 150 to 200 cc. To this solution add from 1 to 2 grams of oxalic acid and sufficient hydrochloric acid to clear the solution. Heat to boiling and neutralize with ammonium hydroxid, stirring constantly. Add ammonium hydroxid in slight excess, and allow to stand for 3 hours, preferably in a warm place. Filter off the supernatant liquid and wash precipitate by decantation once or twice with cold water. Dissolve precipitate with hydrochloric acid, dilute to from 100 to 200 cc, add a little oxalic acid, and precipitate as before. After allowing this precipitate to stand 3 hours, filter, wash with cold water (usually two or three washings are sufficient; owing to the solubility of strontium oxalate excessive washings give low results), dry, ignite and blast, and weigh as calcium and strontium oxids.

Dissolve these oxids with dilute nitric acid and test with the spectroscope for strontium. If strontium is found, transfer the nitric acid solution to a small Erlenmeyer flask and evaporate to complete dryness on the steam bath. Heat at 150° to 160° C. for one or two hours until perfectly dry. Cool, add from 3 to 5 cc of a mixture of equal parts of absolute alcohol and ether to dissolve the calcium nitrate. Cork the flask and allow to stand for 12 hours with occasional shaking; filter, wash with alcohol-ether mixture until a few drops of the filtrate evaporated on platinum foil or watch glass leave no residue. Dry the paper and precipitate; dissolve the strontium nitrate with a few cc of hot water. Add a few drops of sulphuric acid, then a volume of alcohol equal to the volume of the solution. Allow to stand 12 hours, filter, ignite, weigh as strontium sulphate, and calculate to strontium. Test spectroscopically for absence of calcium, subtract from the total oxids of strontium and calcium the strontium oxid equivalent to the strontium sulphate found. The difference is calcium oxid; calculate to calcium. As a check on the calcium oxid, evaporate to dryness the filtrate from the strontium nitrate; dissolve the calcium nitrate in water, precipitate as oxalate, filter, wash, ignite, and weigh as calcium oxid.

(4) That the colorimetric method for iodin and bromin be further studied.

Approved for further study.

Colorimetric method for iodin and bromin.—Evaporate large quantities of the water to dryness after the addition of sodium carbonate in slight excess of the amount necessary to precipitate calcium and strontium. Boil the residue with distilled water; transfer to filter and wash thoroughly with hot water. Evaporate the alkaline filtrate to dryness, add 2 or 3 cc of water to dissolve the residue and enough absolute alcohol to make the percentage of alcohol about 90; boil and filter and repeat the extraction of the residue with alcohol once or twice. Add 2 or 3 drops of sodium hydroxid to the combined alcohol filtrates and evaporate to dryness. Dissolve the residue with 2 or 3 cc of water and repeat the extraction with alcohol as before. Add a drop of sodium hydroxid to the filtrate and evaporate to dryness. Dissolve the residue in a little water, acidify with sulphuric acid (1 to 5), using 3 or 4 drops in excess, and transfer to a small flask. Add 4 drops of 2 per cent potassium nitrite (or sodium nitrite) solution and about 5 cc of carbon bisulphid freshly purified by distillation. Shake until all iodin is extracted, filter off the acid solution from the carbon bisulphid, wash the flask, filter and wash contents with cold distilled water, and transfer the carbon bisulphid (containing the iodin solution) to Nessler tubes, using approximately 5 cc of pure carbon bisulphid. In washing the filter make the contents of the tube up to definite volume, usually 12 or 15 cc, and match the color with that of other tubes containing known amounts of iodin dissolved in carbon bisulphid. Prepare the standard tubes by taking measured quantities of a solution of known potassium iodid content, acidifying with sulphuric acid (1 to 5), adding 3 or 4 drops of potassium nitrite solution and extracting with carbon bisulphid, as in the actual determinations. Transfer to a small flask the sample and standards from which the iodin has been removed. To the standards add definite measured quantities of bromid solution of known strength, and to each of the flasks containing sample and standards add 5 cc of purified carbon bisulphid. Add saturated chlorin water, 1 cc at a time, shaking after each addition until the bromin is all set free. (Care must be taken not to add too much chlorin in excess of that necessary to set the bromin free, since a bromo-chlorid may be formed with an excess of the reagent, thus spoiling the color reaction.) Filter off the water solution from the carbon bisulphid through a moistened filter, wash the contents of the filter two or three times with water, and then transfer to a Nessler tube by means of about 1 cc of carbon bisulphid. Repeat this extraction of the filtrate twice, using 3 cc of carbon bisulphid each time. The combined carbon bisulphid extracts usually amount to from 11.5 to 12 cc. Add enough carbon bisulphid to the tubes to bring them to a definite volume, usually 12 to 15 cc, and compare the sample with the standards. In some cases when working with the method near its upper limit the bromin is not all extracted by the amounts of carbon bisulphid recommended. If so, make one or two extra extractions with carbon bisulphid, transfer the extracts to another tube, and compare the color with some of the lower standards.

(5) That the methods for analysis of water as published in Circular 52, Bureau of Chemistry, and modified in the Proceedings for 1911 be adopted as official, except the methods for strontium, bromin, and iodin.

Approved for final action as official in 1913.

SEPARATION OF NITROGENOUS BODIES (VEGETABLE PROTEINS).

It is recommended—

(1) That the recommendations of the referee concerning the organization of further work upon methods for the analysis of vegetable proteins be referred to a special committee of three, to be appointed by the Chair, to report upon the practicability of organizing in a special way for the continuous and extended study of the subject.

Approved.

COMMITTEE B.—E. M. CHACE, CHAIRMAN.

(Dairy products, foods and feeding stuffs, sugar, tannin, and medicinal plants and drugs.)

DAIRY PRODUCTS.

It is recommended—

(1) That the subjects now under consideration be given further study.

Approved for further study.

FOODS AND FEEDING STUFFS.

It is recommended—

(1) That the method for determining acidity proposed by the previous referee (Bul. 152, p. 198) be adopted as a provisional method.

Finally adopted as provisional.

Acidity of feeds.—Weigh 10 grams of the sample into a shaking bottle, add 200 cc of distilled water, and shake for 15 minutes. Filter the extract through a folded filter and take an aliquot of 20 cc (equal to 1 gram of the sample) for the titration. Dilute with 50 cc of distilled water and titrate with standard decinormal sodium hydroxid solution, using phenolphthalein as indicator.

(2) That the acidity of feeding stuffs be reported in terms of decinormal sodium hydroxid required for neutralization, and that this method of reporting results be adopted as provisional.

Approved for final action as provisional in 1913, although recommended in 1911.

(3) That the petroleum ether method for the determination of fat, especially the comparison of the effects of petroleum ether derived from different crude petroleums, the rate of extraction, and the effect of moisture in the samples (Bul. 152, p. 198; Cir. 90, p. 8) be further studied.

Approved for further study.

(4) That in the petroleum ether method for fat the referee's recommendation that the word "distilling" be substituted for "boiling," making the method read "Extract 2 to 5 grams of the sample without previous drying for 3 hours in a Soxhlet apparatus with petroleum ether distilling below 65° C.," be not adopted.

Recommendation of committee adopted.

(5) That an associate referee be appointed to study the questions of the proper factor for the conversion of nitrogen into protein, and the acidity in feeding stuffs with special reference to eliminating the error due to proteins if these problems are of sufficient importance.

No action.

SUGAR.

It is recommended—

(1) That the modified Clerget and the direct sucrose methods be tested further.

Approved for further study.

(2) That the resolutions of the International Commission for Uniform Methods of Sugar Analysis of the Eighth International Congress of Applied Chemistry concerning the temperature of polarizations and the use of a neutral potassium bichromate light filter cell be referred to the referee on sugar for a report at the next meeting.

Approved.

(3) That to the official aerometric method for the determination of total solids (Bul. 107, Rev., p. 65) a note be added to the effect that the results on molasses and other materials containing large amounts of invert sugar or nonsugar solids are only roughly approximate.

Not considered. (Available for final action in 1913.)

MEDICINAL PLANTS AND DRUGS.

It is recommended—

(1) That the subjects now under consideration be given further study.

Approved for further study.

COMMITTEE C.—P. F. TROWBRIDGE, CHAIRMAN.

(Food adulteration and separation of nitrogenous bodies (meat proteins).)

COLORS.

It is recommended—

(1) That the paper on the examination for coloring matter by the referee be given further study with a view to its adoption as a provisional method in 1913.

Approved for final action as provisional in 1913.

SACCHARINE PRODUCTS.

It is recommended—

(1) That the method for the determination of solids in molasses and other sugar products, by means of the refractometer, using Geerlig's table of equivalents and temperature corrections, be adopted as provisional, but that the results be expressed as percentages calculated from the refractometer readings.

Approved for final action as provisional in 1913.

(2) That the suggestions of the referee for 1911 as to further study be adopted.

Approved.

FRUIT PRODUCTS.

It is recommended—

(1) That a general method of procedure based upon the uranium acetate method of Yoder be tried in the estimation of malates in cane and maple products during the coming year.

Approved.

(It is hoped that this general method will permit of a determination of malic acid in fruit products containing much color and also tartaric acid.)

VINEGAR.

It is recommended—

(1) That in method "8. Reducing sugars before inversion after evaporation." as printed in the 1911 Proceedings (Bul. 152, p. 126), the words "Again add 25 cc water and evaporate to 5 cc" be omitted, the method then reading as follows: "Evaporate 50 cc to 5 cc on the water bath. Add 25 cc of water and evaporate to 5 cc. Transfer to a volumetric flask, make up to the mark, and proceed as under method 7, using a quantity equivalent to 10 or 20 cc of sample," and that this be considered for adoption as provisional in 1913.

Approved for final action as provisional in 1913.

(2) That the method for polarization (Bul. 152, p. 126) be studied with reference to whether or not the true polarization is obtained after clarifying with lead.

Approved for further study.

(3) That Fincke's method for formic acid be studied during the coming year with a view to final adoption as provisional in 1913.

Approved for final action as provisional in 1913.

Fincke's method for formic acid (Zts. Nahr. Genussm., 1911, *21*:1; *22*:88).—Add 0.4 to 0.5 gram of tartaric acid to 100 cc of sample and distill with steam. Pass the outflowing stream through a boiling mixture of 15 grams of calcium carbonate and 100 cc of water and keep this volume constant throughout the process. Collect 1,000 cc of distillate and reduce the volume of sample to 30 to 40 cc. Discard the distillate. Filter and wash the calcium carbonate mixture, make faintly acid with hydrochloric acid, add 10 to 15 cc of mercuric chlorid solution (10 grams of mercuric chlorid and 3 grams of sodium chlorid to 100 cc), and heat in a boiling water bath for two hours. Filter on a tared Gooch crucible and wash with water, alcohol, and finally ether. Dry and weigh as mercurous chlorid. Factor for formic acid is 0.0977.

(4) That methods 6, 11, 15, 16, and 17, as printed in the 1911 Proceedings, be further studied.

Approved for further study.

6. *Solids.*—Measure 10 cc of filtered vinegar into a tared flat-bottom platinum dish of 50 mm diameter, evaporate on the water bath to a thick sirup, and dry for exactly two and one-half hours in the drying oven at the temperature of boiling water; cool and weigh. It is essential to use a flat-bottom dish.

11. *Ash.*—Measure 25 cc into a tared platinum dish, evaporate to dryness on the steam bath, heat in the muffle at low heat to expel inflammable gases, treat the charred portion with a few cubic centimeters of water, and evaporate dry on the bath; replace in the muffle at low redness for 15 minutes, and continue the alternate evaporation and heating until a white or gray ash is obtained, at no time allowing the temperature to exceed a dull red; cool in desiccator and weigh.

15. *Fixed acid.*—Measure 10 cc into a 200-cc porcelain casserole, evaporate just to dryness, add 5 to 10 cc of water, and again evaporate; repeat until at least five evaporations have taken place and no odor of acetic acid can be detected. Add nearly 200 cc of recently boiled distilled water and titrate with tenth-normal alkali, using phenolphthalein. One cubic centimeter of tenth-normal alkali is equivalent to 0.0067 gram of malic acid.

16. *Volatile acid.*—Calculate the fixed acid as acetic and deduct from the total acid. Express as acetic acid.

17. *Lead precipitate.*—To 10 cc in a test tube, add 2 cc normal lead acetate (20 per cent solution), shake, and let stand one-half hour. Express as turbidity, light, medium, heavy, or very heavy.

(5) That methods 1, 2, 3, 4, 7, 9, 12, and 18, as printed in the 1911 Proceedings, be adopted as provisional.

Approved for final action as provisional in 1913.

1. *Preparation of sample.*—For microscopical examination employ the original sample, but for chemical analysis filter if turbid.

2. *Calculation of results.*—Express all results as grams per 100 cc.

3. *Specific gravity.*—Determine as directed under XIII, Wine, page 83, Bulletin 107, Revised.

4. *Alcohol.*—Measure 100 cc of the sample into a round-bottom distilling flask. Make faintly alkaline with saturated caustic soda solution, add a small scrap of paraffin, distill almost 50 cc, make up to 50 cc at temperature of sample, filter if necessary, and determine specific gravity by pyknometer or Sprengel tube. Calculate per cent by volume, or grams per 100 cc, from Table II, page 203, bearing in mind that the alcohol strength of the distillate is twice that of the original vinegar.

7. *Total reducing matters before inversion.*—Proceed according to Munson and Walker's method (Bul. 107, Rev., p. 241), using 10 or 20 cc of sample.

Express results as grams of invert sugar per 100 cc. Malt vinegar should be clarified with sodium phosphotungstate.

9. *Reducing sugar after inversion.*—Proceed as under "8." After the last evaporation to 5 cc transfer to a 100 cc flask with 70 cc of water and invert by one of the methods given under "VI. General methods (c)," page 40. Nearly neutralize with caustic soda, make up to the mark, and proceed as under "7," using a quantity equivalent to 10 or 20 cc of sample.

12. *Solubility and alkalinity of soluble ash.*—Add to the above ash about 10 to 15 cc distilled water, bring to a boil, and filter through a 9 cm quantitative filter. Repeat the operation twice; transfer the ash completely to the filter paper and wash with three successive portions of hot water; dry and ignite the filter with the undissolved residue at low red heat; cool, weigh, and calculate as insoluble ash. Cool the filtrate and titrate with tenth-normal hydrochloric acid, using methyl orange as indicator. Express results as number of cubic centimeters of tenth-normal hydrochloric acid per 100 cc sample.

18. *Color—Brewer's scale.*—Read in good, reflected daylight, using 0.5-inch cell and the Lovibond scale.

(6) That the method for the determination of glycerin (Bul. 137, pp. 61–63) be adopted as provisional.

Finally adopted as provisional.

FLAVORING EXTRACTS.

It is recommended—

(1) That the method for the detection of capsicum in ginger extract (Cir. 90, p. 13), as proposed by Doyle, modifying the La Wall method, be adopted as provisional. (The Doyle method is not essentially different from La Wall's. The details of procedure, however, are such as to make the test more positive and are set forth more clearly than in the latter method.)

Finally adopted as provisional.

(2) That the limits of composition of standard vanilla extracts be referred to the Committee on Food Standards for such action as they think advisable.

Approved.

(3) That the study of the composition of pure commercial vanilla extracts be continued next year for the purpose of securing additional data.

Approved.

(4) That Wichmann's method for detecting coumarin (Cir. 95) be further studied.

Approved for further study.

(5) That the study of the method (Bul. 137, p. 79) for the examination of ginger extracts and other flavoring materials, as recommended last year, be made the subject of further work next year.

Approved.

SPICES.

It is recommended—

(1) That an effort be made to devise methods other than microscopical for detecting an excess of seeds in paprika.

Approved.

(2) That, if possible. samples of prepared mustard of known composition be submitted to collaborators for determination of crude fiber by the present official methods.

Approved.

BAKING POWDER.

It is recommended—

(1) That the study of methods for detecting arsenic and lead in baking powder ingredients be continued.

Approved.

MEAT AND FISH.

It is recommended—

(1) That on page 106 of Bulletin 107, Revised, under "XVII. Methods for the analysis of meat and meat products. 1. Identification of species—Provisional," fourth line, after "melting point," "melting point of stearin by Belfield-Emery method" be inserted.

Approved for final action as provisional in 1913.

(2) That Mayerhofer's method, modified (Bul. 107, Rev., p. 109), and Price's method for the detection or determination of starch be studied during the next year.

Approved for further study.

Price's method.—In a 200-cc beaker treat 10 grams of finely divided meat with 75 cc of 8 per cent solution of potassium hydrate in 95 per cent alcohol, and heat on the steam bath until all the meat is dissolved. This will take from 30 to 45 minutes. Add an equal volume of 95 per cent alcohol, cool, and allow to stand at least one hour. Filter by suction through a thin layer of asbestos in a Gooch crucible or through an alundum crucible. Wash twice with warm 4 per cent potassium hydrate in 50 per cent alcohol and then twice with warm 50 per cent alcohol. Endeavor to retain as much of the precipitate in the beaker as possible until the last washing. Place the crucible with contents in the original beaker, add 40 cc water and 25 cc concentrated sulphuric acid. Stir during the addition of the acid and see that the acid comes in contact with all the precipitate. Allow to stand about 5 minutes, add 40 cc of water, and heat just to boiling, stirring constantly. Transfer the solution to a 500-cc graduated flask, add 2 cc of a 20 per cent solution of phosphotungstic acid, allow to cool to room temperature, and make up to mark with distilled water. Filter through a starch-free filter paper and after neutralizing determine the dextrose present in a 50 cc portion of the filtrate with Fehling's solution, using Low's method, Bulletin 107, Revised, page 241, for the determination of the copper in the cuprous oxid precipitate. Dextrose times 0.9 equals starch.

(3) That Folin's method by aeration, as modified, be substituted for the method for ammoniacal nitrogen in animal substances under "(e) Ammonia.—Provisional," Bulletin 107, Revised, page 115.

Approved for final action as provisional in 1913.

Folin's method, modified.—Arrange 5 vessels in series as follows: (1) A bottle containing sulphuric acid, with a Hopkins safety bulb, to purify the entering air. (2) A 1,000-cc flask containing 25 grams of sample, 250 cc of water, 5 grams of sodium chlorid, and 1 gram of sodium carbonate. Alcohol may be added to prevent foaming. (3) A 250-cc safety flask. (4) A cylinder, fitted with a Folin absorption tube, containing tenth-normal sulphuric acid. (5) A 100-cc safety flask.

The last flask should be connected with an air pump powerful enough to draw the ammonia over into the standard acid. Alcohol may be substituted almost wholly for the water, if the air current is weak. Titrate the standard acid at intervals of an hour until no more ammonia is given off, running a blank at the same time. Methyl red, cochineal, or congo red may be used in aqueous solutions, methyl red or cochineal in alcoholic.

(4) That the foaming difficulty of the aeration method be further studied.

Approved for further study.

(5) That the following distillation method be studied: Mix 25 grams of the fine sample with 5 grams of salt and 1 gram of sodium carbonate, and 100 cc of alcohol, in a 500-cc flask, and pass through it a current of vapor from boiling alcohol. Distill a 200-cc portion and titrate and then treat similarly two 100-cc portions. The last portion should contain very little ammonia.

Approved for study.

FATS AND OILS.

It is recommended—

(1) That the Emery method for the detection of added beef fat in lard (U. S. Dept. Agr., Bureau of Animal Industry Cir. 132), reported at the 1911 meeting, be made provisional.

Approved for final action as provisional in 1913.

(2) That final action be taken to make the provisional method for the preparation of samples (Bul. 107, Rev., p. 129) official.

Finally adopted as official.

(3) That method "(c) Zeiss Butyro-Refractometer" (Bul. 107, Rev., p. 132) be finally adopted as official.

Adopted, final action.

(4) That method "12. Free Fatty Acids.—Provisional" (Bul. 107, Rev., p. 142) be finally adopted as official.

Adopted, final action.

(5) That the Halphen reaction for cottonseed oil (Bul. 107, Rev., p. 144, 17 (b)) be finally adopted as official.

Adopted, final action.

(6) That the Baudouin test for sesame oil (Bul. 107, Rev., p. 146, 17 (e)) be finally adopted as official.

Adopted, final action.

(7) That the Villavecchia test for sesame oil (Bul. 107, Rev., p. 146, 17 (f)) be finally adopted as official.

Adopted, final action.

(8) That any cut appearing in the text of the chapter on fats and oils, Bulletin 107, Revised, be considered merely as an illustration and not as an integral part of the method.

Adopted, final action.

(9) That 75° in addition or instead of 100° be adopted provisionally with a view to ultimately making it official for the determination of the specific gravity of high melting point fats.

Approved for final action as provisional in 1913.

(10) That the glycerin saponification method for the preparation of fatty acids for use in the titer test, as given in the associate referee's report, be adopted as provisional.

Approved for final action as provisional in 1913.

Glycerol method.—Heat to 150° C. in an 800-cc beaker, 75 cc of a glycerol potassium hydroxid solution, made by dissolving 25 grams of potassium hydroxid in 100 cc of high-test glycerol; then add 50 cc of the oil or melted fat, previously filtered if necessary to remove foreign substances. Saponification in many cases takes place almost immediately, but heating, with frequent stirring, should be continued for 15 minutes, care being taken that the temperature does not rise much above 150° C. When the saponification is complete, as indicated by the perfectly homogeneous solution, pour the soap into an 800-cc casserole containing about 500 cc of nearly boiling water, carefully add 50 cc of 30 per cent sulphuric acid, and heat the solution, with frequent stirring, until the layer of fatty acids separates out perfectly clear. Transfer the fatty acids to a tall separatory funnel, wash three or four times with boiling water to remove all mineral acids, draw them off into a small beaker, and allow to stand on a steam bath until the water has settled out and they are clear. Filter into a dry beaker and heat to 150° C. on a thin asbestos plate, stirring continually with the thermometer, transfer to a titer tube, filling it to within 1 inch of the top, and take the titer as indicated in the present provisional method (Bul. 107, Rev., p. 135).

(11) That action on the Bechi or silver nitrate test for cottonseed oil as an official method be deferred.

Approved.

DAIRY PRODUCTS.

It is recommended—

(1) That the method proposed in 1911 (Bul. 152, p. 101; Cir. 90, p. 10) as applied to milk, evaporated milk, sweetened condensed milk, thin cream, and ice cream, be given further study.

Approved for further study.

(2) That for rich cream the following modification be given further study: Add 50 grams of the material, stirring vigorously, to 25 cc boiling Soxhlet solution, in a 250-cc beaker. Cover the filter with a thin layer of fibrous asbestos mixture, carefully covering the sides as far up as possible. Wash once or twice with cold water, and proceed as in the original method.

Approved for further study.

CEREAL PRODUCTS.

It is recommended—

(1) That the method of Bryan, Given, and Straughn (Cir. 71) for soluble carbohydrates be given a further trial.

Approved for further study.

(2) That methods for the estimation of moisture by the use of the vacuum oven and vacuum desiccator, for estimating the acidity of the water extract of flour, and Olsen's method for dry gluten be referred to the next referee for immediate consideration.

Approved for study.

(3) That the methods for the determination of nitrous nitrogen (Bul. 152, p. 113) be finally adopted as provisional.

Finally adopted as provisional.

(4) That method B for the determination of ash (Cir. 90, p. 12) be made a provisional method.

Not considered. (Available for final action as provisional in 1913.)

(5) That the method for the determination of ether extract given in Bulletin 107, Revised, page 39, 5, (b), (1), be made official.

Not considered. (Available for final action as official in 1913.)

CONDIMENTS OTHER THAN SPICES.

It is recommended—

(1) That the methods for the determination of lactic acid, citric acid, insoluble solids, and sand be made provisional.

Approved for final action as provisional in 1913.

Lactic acid.—Transfer 100 grams of the sample to a 500-cc bottle suitable for use in the centrifuge. Dilute to about 450 cc with water and add 10 cc of 20 per cent normal lead acetate. Shake well and centrifuge. Test supernatant liquid with a drop of lead acetate solution to determine whether precipitation is complete. If necessary add a few cubic centimeters more lead acetate solution, shake again, and centrifuge. When precipitation is complete dilute to 500 cc with water, mix well, centrifuge, and filter. Measure out the largest possible aliquot of the filtrate (usually 400 cc) and add a slight excess of sulphuric acid. Filter, wash the precipitated lead sulphate with a small amount of water, and evaporate the filtrate on the steam bath to a convenient volume for extraction in a continuous liquid extractor (usually 100 cc), extract for from 18 to 20 hours in a liquid extractor with washed ether (alcohol free). In case the quantity of lactic acid present is greater than 0.5 gram it is usually necessary to extract for a longer period. In any case it is best to reextract for from 8 to 10 hours to make sure that the extraction is complete. (Ether sufficiently pure for this purpose may be prepared by shaking out ordinary ether once with a sodium hydrate solution and then 10 times with small quantities of water.) Transfer the ether extract to a beaker, add about 20 cc of water, and evaporate the ether on the steam bath. Continue the evaporation until only about 10 cc of solution remains. Dilute to 20 cc and again evaporate to 10 cc to remove all traces of ether. Filter if necessary. To the clear solution add approximately 3 grams of sodium hydrate and exactly 50 cc of a 1½ per cent solution of potassium permanganate. Heat the mixture on a water bath at 100° C. for half an hour. At the end of that time the solution should have a decided blue-black or purple color. If the solution turns green or colorless above a layer of brown precipitate at any time during the course of the oxidation, more standard permanganate in measured portions must be added until the blue-black or purple color remains throughout a half hour of heating. The oxidation is then complete. Strongly acidify the hot solution with about 50 cc of 10 per cent sulphuric acid and run in standard permanganate from a burette until the solution is decolorized. A 5 per cent solution of oxalic acid is convenient for this purpose. Titrate back any slight excess of oxalic acid with the standard permanganate solution. Any standard solutions of potassium permanganate and oxalic acid may be used for these titrations within reasonable limits of strength. In alkaline solution the permanganate oxidizes the lactic acid quantitatively to oxalic acid according to the equation:

$$2C_3H_6O_3+10KMnO_4=2(COOH)_2+4H_2O+2CO_2+5MnO_2+5K_2MnO_4.$$

Then, in acid solution, the oxalic acid is further oxidized by the permanganate to carbon dioxid and water according to the equation:

$$5(COOH)_2+2KMnO_4+3H_2SO_4=10CO_2+8H_2O+K_2SO_4+2MnSO_4.$$

Calculation: The total weight of permanganate used in the oxidation of the lactic acid is determined by subtracting the permanganate equivalent of the oxalic acid used from the total amount of permanganate added. The weight of permanganate times 0.237 equals the weight of lactic acid.

Citric acid.—Weigh 25 grams of the sample into a 250-cc beaker, dilute to approximately 200 cc with 95 per cent alcohol, allow to stand, with frequent stirring, for 4 hours, filter through a folded filter, and wash with 50 cc of 80 per cent alcohol. To the filtrate add sufficient water to dilute the alcohol to 50 or 60 per cent, then add 10 cc of 20 per cent barium acetate solution. Stir well with a glass rod and allow to stand overnight. In the morning filter on a Gooch crucible, wash with 50 per cent alcohol, dry for 3 to 4 hours in the oven at 100° C., and weigh. Multiply the weight of precipitate by the empirical factor 0.51 to obtain the weight of anhydrous citric acid.

Insoluble solids.—Wash 20 grams repeatedly with hot water, centrifuging after each addition of water and pouring the clear supernatant liquid through a tared triple filter paper on a Büchner funnel. After 4 or 5 washings transfer the remaining insoluble matter to the filter, dry for 2 hours at 100° C., cool in a desiccator, and weigh rapidly in the air. A triple filter paper is used, as strong suction will usually rupture a single or double layer of paper. The paper used should be dried for 2 hours at 100° C., cooled in a desiccator, and the original weighing made rapidly in the air. A cylinder 1 to 1¼ inches in diameter and 5 to 6 inches long is convenient for washing and centrifuging. This may be prepared by shortening a colorimeter tube.

Sand.—Weigh 100 grams of well-mixed sample into a 2 or 3 liter beaker, nearly fill the beaker with water, and mix the contents thoroughly. Allow to stand 5 minutes and decant the supernatant liquid into a second beaker. Refill the first with water and again mix the contents. After 5 minutes more decant the second beaker into a third, the first into the second, refill, and again mix the first. Continue this operation, decanting from the third beaker into the sink until the lighter material is washed out from the catsup. Then collect the sand from the 3 beakers into a tared Gooch crucible, dry, ignite, and weigh. Attention is especially called to the fact that under " Sand " only the figure obtained by this method should be reported. The results obtained by the determination of ash insoluble in hydrochloric acid are not applicable to the determination of sand, since the sand is so unevenly distributed that reliable results can only be obtained by taking a larger sample than is possible in the determination of ash.

(2) That the committee on editing the new edition of the methods of analysis make provision for minor changes suggested by the referee in methods for the determination of total solids, soluble solids, ash, alkalinity of ash, sodium chlorid, reducing sugars before inversion, reducing sugars after inversion, sucrose, polarization after inversion, total acids as citric, volatile acids as acetic, fixed acids as citric, and detection of butyric acid, so that they may be used as optional methods.

Approved.

(3) That the Boyle method for the determination of turmeric in prepared mustard be given trial.

Approved for study.

Boyle method.—(Preparation of samples of prepared mustard with known amounts of turmeric to serve as standards.) Rub 5-gram samples of the standards and a 5-gram sample of the mustard under examination in a mortar with 50 cc of 95 per cent alcohol and transfer to 100-cc flasks. Shake these flasks occasionally during about one hour, make up to the mark with 95 per cent alcohol, and filter. Place 1 cc of each filtrate in a 50-cc beaker, hang a strip of filter paper about 1 inch wide in the beaker so that it just touches the bottom. The alcoholic solution is then absorbed by the paper, which is allowed to dry in the air. The depth of color is then compared with the standards.

COCOA AND COCOA PRODUCTS.

It is recommended—

(1) That the method for the determination of casein in milk chocolate as studied by the association this year (Bul. 152, p. 163) be adopted as provisional.

Approved for final action as provisional in 1913.

(2) That the method of determining milk fat in milk chocolate (Bul. 152, p. 159). as studied by the association for two years and as reported this year, be adopted as provisional.

Approved for final action as provisional in 1913.

(3) That the methods for crude starch in sweet and unsweetened products be further studied by the referee next year.

Approved for further study.

(4) That petroleum ether be substituted for sulphuric ether as a solvent in the determination of fat in cocoa products. The committee recommend that this be not adopted.

Recommendation of committee adopted.

TEA AND COFFEE.

It is recommended—

(1) That the referee for the coming year continue the lines of work as laid down by previous referees.

Approved.

PRESERVATIVES.

It is recommended—

(1) That the methods of qualitative and quantitative estimation of formic acid be further worked out, and that the mercuric chlorid method be carefully studied to determine its accuracy.

Approved for study.

WATER IN FOODS.

It is recommended—

(1) That further study be made of the vacuum method (Bul. 122, p. 219) of determining moisture in food, using different dehydrating agents.

Approved for further study.

ORGANIC AND INORGANIC PHOSPHORUS IN FOODS.

It is recommended—

(1) That the referee for the coming year continue the lines of work as laid down by previous referees.

Approved.

HEAVY METALS IN FOODS.

It is recommended—

(1) That a further study be made of the various modifications of the nitric-sulphuric acid method for destroying organic matter in the determination of tin in foods.

Approved for further study.

(2) That the use of potassium hydroxid, in place of yellow ammonium sulphid, in the solution of tin sulphids be further investigated.

Approved for further study.

(3) That a study be made of the electro deposition of tin, especially from sulphid solutions.

Approved for study.

(4) That the volumetric method of the American Can Co., applicable to the determination of tin in foods, be made the subject of cooperative work.

Approved.

Volumetric method of the American Can Co.—Tin after wet combustion is precipitated in the usual way by hydrogen sulphid. Filter this precipitate directly on a Gooch crucible with a false bottom and wash; this is very quickly accomplished under suction. Then transfer the precipitate with the asbestos pad simply by pushing in the false bottom of an Erlenmeyer flask; digest with the addition of hydrochloric acid and potassium chlorate; dispel the chlorin by the addition of some aluminum foil. Then attach the flasks in duplicate to a Kipp apparatus producing carbon dioxid. Raise the delivery tubes from the water seal out of the water and allow the gas to run through the Erlenmeyer flask for a few minutes. Then drop the delivery tubes to the bottom of the water seal cylinder and put two pieces of aluminum foil, weighing probably a gram or a gram and a half, into the Erlenmeyer flasks. The whole is put into solution, of course not including the asbestos. Cool the flasks in ice water and disconnect them one at a time; wash out the bulbs with the air-free water and dilute so that the hydrochloric acid will not be over about one-third strength. Then titrate as quickly as possible with one-hundredth normal iodin, using starch paste as an indicator.

If it is desired to titrate by the excess method, an excess of one-hundredth normal iodin diluted with the proper amount of water to take care of the acidity in the flask is added directly while the flasks are under carbon dioxid insulation, by lifting up the cork and pouring in the iodin solution while the carbon dioxid is running out and preventing any contact of the air with the solution. The excess of iodin is then titrated back with the one-hundredth normal thiosulphate.

(5) That the use of a Gooch crucible in filtering the first tin sulphid precipitate in the gravimetric methods be tried.

Approved.

DISTILLED LIQUORS, WINE, AND BEER.

It is recommended—

(1) That the referees for the coming year continue the lines of work as laid down by previous referees.

Approved.

SEPARATION OF NITROGENOUS BODIES (MEAT PROTEINS).

I. Meats and Beef Extracts.

It is recommended—

(1) That in Bulletin 107, Revised, page 108, 7 (a), the following sentence be added to the Kjeldahl method and recognized as provisional: "If desired, 5 to 7 grams of potassium sulphate may be added in addition to the mercury of the Kjeldahl method, and no potassium permanganate be used." This modification should be designated as the Kjeldahl-Gunning-Arnold method.

Approved for final action as provisional in 1913.

(2) That the length of time of digestion with the Kjeldahl-Gunning-Arnold method be studied further, with a view of ascertaining whether 1½ hours' digestion after clearing up is as complete as when digestion is carried on 4 hours by the Kjeldahl or Gunning method.

Approved for further study.

II. NITROGENOUS BODIES IN MEATS.

It is recommended—

(1) That in Bulletin 107, Revised, page 108, 7 (b), the following method be studied during the coming year with the idea of its being made optional for determining insoluble protein: Exhaust 7 to 25 grams of the sample (depending upon its moisture content) with 330 cc of cold (15° C.) distilled water by making 11 successive extractions, 4 of 50 cc, 4 of 25 cc, and 3 of 10 cc each. Make extract up to 500 cc and determine total soluble nitrogen in 50 cc. Deduct percentage of soluble nitrogen from the total and multiply difference by 6.25 for insoluble protein.

Approved for final action in 1913.

(2) That in connection with recommendation II (1) a comparative study be made of the method as given in Bulletin 107, Revised, page 108, 7 (d), with the following method: Evaporate 150 cc of filtrate from II (1) to about 40 cc and nearly neutralize to litmus (paper), having it faintly acid. Heat on steam bath 5 minutes. Filter, wash, transfer paper and contents to flask, and determine nitrogen as "total nitrogen." In both methods use the same volume of the filtrate from II (1) and bring to same neutrality.

Approved.

(3) That further study be made of the method for determining creatin and creatinin in meat and beef extract, with the idea of referring it to the association for final action in 1913.

Approved for final action in 1913.

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  Determination of nitrogen: C. L. Hare, Auburn, Ala.
  Separation of nitrogenous bodies: A. D. Emmett, Urbana, Ill.
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Phosphoric acid: L. S. Walker, Amherst, Mass.
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  Determination of nitrogen: To be appointed.
  Separation of nitrogenous bodies—
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    Vegetable proteins: T. B. Osborne, New Haven, Conn.
    Meat proteins: A. D. Emmett, Urbana, Ill.
Potash:
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Recommendation of Referees and Revision of Methods.

(Figures in parentheses refer to year in which appointment expires.)

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SUBCOMMITTEE B: F. W. Woll (1918), P. F. Trowbridge (1916), *E. M. Chace (1914), chairman, Bureau of Chemistry, United States Department of Agriculture, Washington, D. C.* (Dairy products, foods and feeding stuffs, sugar, tannin, medicinal plants and drugs, water in foods, separation of organic and inorganic phosphorus in foods, separation of nitrogenous bodies.)

SUBCOMMITTEE C: L. M. Tolman (1918), *H. E. Barnard (1916), chairman, Indianapolis, Ind.*, C. D. Howard (1914). (Food adulteration.)

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Approved:
JAMES WILSON,
Secretary of Agriculture.
WASHINGTON, D. C., *October 22, 1912.*

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